Mesoporous Carbon Xerogel Synthesis and Activation by Oxidative Treatment

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Abstract

Mesoporous carbon xerogels have drawn a lot of interest as adaptable materials because of their distinctive structural and surface characteristics. The creation of mesoporous carbon xerogels and the subsequent oxidative activation of such materials are the main topics of this research study. These treatments adjust the surface's chemical properties, such as acidity and basicity, providing chances for customisation to fit certain applications. This study examines the synthesis process, the impact of surface oxidation on surface characteristics, and the prospective uses of activated carbon xerogels.

Keywords: Carbon catalysts, Surface acidity, Oxidative activation, and Carbon xerogel.

INTRODUCTION

Mesoporous carbon xerogels have gained prominence as very promising materials with a broad variety of uses in areas including energy storage, catalysis, sensing, and environmental remediation. These materials are excellent prospects for specialised applications due to their distinctive structural qualities, which include a large surface area, a well-defined pore structure, and variable surface chemistry.

The performance and utility of carbon xerogels in various applications are greatly influenced by the surface chemical characteristics of these materials. Their catalytic activity, adsorption capacity, and interactions with target molecules are all impacted by the presence of acidic and basic sites on the surface. Therefore, it is crucial to be able to regulate and adjust the surface chemistry of carbon xerogels in order to maximise their functionality for particular applications.

Oxidative treatment is a useful technique for modifying the surface chemical properties of carbon xerogels. In oxidative treatments, the xerogels are exposed to different oxidising substances including air, nitric acid, or sulfuric acid, which cause chemical changes on the surface. These modifications change the concentration of acidic and basic sites, adjust the surface oxygen groups, and introduce or enhance certain functional groups.

Mesoporous carbon xerogel synthesis

Starting with the sol-gel condensation of resorcinol and formaldehyde, a succession of precisely regulated processes are required to create mesoporous carbon xerogel. The main steps of the

synthesis process, such as pH adjustment, gel curing, grinding, washing, drying, and carbonization, are described in this section.

Formaldehyde and Resorcinol Sol-Gel Condensation

Due to its simplicity and adaptability, the sol-gel condensation process is often used for the synthesis of carbon xerogels. Resorcinol and formaldehyde are employed as precursors in this method. Formaldehyde serves as a carbon source, while resorcinol works as a cross-linking agent.

Resorcinol and formaldehyde go through polymerization processes in an aqueous solution during the sol-gel condensation. To produce a carbon xerogel with the appropriate characteristics, the formaldehyde/resorcinol ratio is carefully regulated. Usually, a formaldehyde/resorcinol ratio of 2.0 is used, however adjustments may be made depending on the situation.

pH Modification and Gel Curing Process

To achieve ideal gel formation and stability during sol-gel condensation, the pH of the gel is adjusted. A sodium hydroxide (NaOH) solution that has been diluted is added to make this correction. The final carbon xerogel's porosity and gel structure are greatly influenced by the pH level. The pH of the sol-gel procedure in this synthesis is set to 5.7 using diluted NaOH solution.

The organic gel is exposed to a curing procedure after pH correction. The gel is put in the proper setting, usually at a moderate temperature (such as 75 °C), and allowed to cure for a certain amount of time, such as 36 hours. The gel may solidify and gain mechanical strength during this curing phase, ready it for further processing.

Steps for Grinding, Washing, and Drying

The gel is crushed or ground to the appropriate particle size after it has fully cured. This procedure seeks to provide an appropriate particle size distribution for processing and application later on. Typically, the recommended size range is between 0.2 mm and 2.0 mm.

The gel particles are properly washed after grinding to get rid of any contaminants or leftover chemicals. To remove the sodium hydroxide employed in the pH adjustment stage, the gel is first repeatedly rinsed with 2 N acetic acid. The leftover acetic acid is then removed by repeatedly washing the gel particles with distilled water.

After being washed, the gel particles go through a drying process to get rid of the liquid. The gel is typically dried in an air oven at a certain temperature (for example, 120 °C) for a predetermined amount of time (for example, 48 hours). The result of this drying procedure is an organic dried gel known as RF xerogel (resorcinol-formaldehyde).

The Process of Carbonization and the Resultant Carbon Xerogel

After that, the dried RF xerogel is put into a quartz tube reactor and put inside a tube furnace. To generate an inert environment, a flow of nitrogen gas is created through the reactor. Up until it reaches the appropriate carbonization temperature (for example, 700 °C), the temperature within the

furnace is steadily raised at a regulated ramp rate (for example, 5 °C min-1).

The RF xerogel undergoes carbonization under the regulated temperature and nitrogen flow, resulting in the elimination of non-carbon components and the production of a carbonaceous structure. To guarantee that the transformation of carbon is complete, this carbonization procedure is often carried out for a predetermined amount of time (for example, 4 hours).

The reactor is gradually cooled to room temperature when carbonization is finished, and the resultant material is carefully collected. The mesoporous carbon xerogel, known in this synthesis as CX-UA, is the substance that was produced at this point. The mesoporous carbon xerogel is excellent for a variety of applications because to its distinct surface chemistry, large surface area, and well-defined porous structure.

Surface Activation Oxidative Treatment

Mesoporous carbon xerogels' performance in diverse applications is greatly influenced by their surface characteristics. By adding oxidative treatments, it is possible to improve the surface chemistry of carbon xerogels and adjust their characteristics for particular applications. The impact of oxidative treatment in surface activation is examined in this section along with the effects of various treatments on the surface characteristics of carbon xerogels.

Oxidative Surface Treatment to Change Surface Properties

To cause chemical reactions on the surface, oxidative treatments entail exposing the carbon xerogels to oxidising substances like acids or air. The surface chemistry may change as a result of these treatments, and new functional groups may develop or existing ones may be modified. Oxidative treatments give a technique to tailor the carbon xerogels for desired applications by selectively changing the surface characteristics.

Surface Acidity and the Effects of Nitric Acid Treatment

The surface of carbon xerogels is often activated by nitric acid (HNO3) treatment. Nitric acid is introduced, and as a result, acidic functional groups are produced, increasing the acidity of the surface. On the carbon xerogel surface, the nitric acid treatment results in the creation of carboxylic acid groups, phenolic groups, and carboxylic anhydride groups. These acidic functional groups contribute to the material's increased acidity, which is advantageous for acid-catalyzed reactions and catalytic applications.

Sulfuric acid treatment's effects on surface basicity

Another oxidative technique utilised for surface activation of carbon xerogels is sulfuric acid (H2SO4) treatment. Sulfuric acid treatment, as opposed to nitric acid treatment, eliminates basic sites from the carbon xerogel surface. No discernible basicity is seen on the surface of the carbon xerogels after the sulfuric acid treatment. The loss of basic functional groups or the conversion of basic sites into acidic or neutral species are credited with this decrease in basicity. The main goal of the sulfuric acid treatment is to make the carbon xerogels more acidic.

Surface Chemistry Modification by Air Oxidation

The surface of carbon xerogels is activated using air oxidation in addition to acid treatments. When materials are exposed to air, oxidative surface processes take place that result in the introduction of functional groups that contain oxygen. These functional groups, which also contribute to the acidity of the surface, include carbonyl/quinone groups. Air oxidation has the tendency to lower the surface basicity while concurrently raising the acidity on the surface. In contrast to acid treatments, air oxidation causes very minor changes in the surface chemistry.

The surface characteristics of carbon xerogels may be selectively altered by using oxidative treatments like nitric acid, sulfuric acid, or air oxidation. Specific functional groups may be introduced, the surface acidity is increased, and basicity is decreased thanks to these treatments. These adjustments enable customised surface characteristics that are ideal for use in catalysis, adsorption, and other related areas. The experimental approaches and characterisation methods utilised to examine the surface characteristics of carbon xerogels activated by oxidative treatments will be covered in more detail in the next parts of this work.

Characterization Techniques

Different characterisation approaches are used to comprehend how oxidative treatment affects the surface characteristics of mesoporous carbon xerogels. These methods provide insightful information on the structure, surface area, pore size distribution, pore volume, surface acidity, and basicity of the activated carbon xerogels. We will go through the characterisation methods often used to examine carbon xerogels in this section.

Pore volume, pore size distribution, and surface area analysis

Analysing the surface area, pore size distribution, and pore volume of carbon xerogels is essential for determining their porosity and adsorption capabilities. Nitrogen adsorption-desorption isotherm analysis utilising the Brunauer-Emmett-Teller (BET) method is one of the extensively utilised methods for this purpose. The carbon xerogels' precise surface area, pore size distribution, and pore volume are all disclosed by the BET study. To further the analysis of pore structure and morphology, methods like mercury porosimetry and scanning electron microscopy (SEM) may also be used.

Surface Acidity and Basicity Measurement

Understanding the surface chemistry and catalytic capabilities of carbon xerogels requires the assessment of surface acidity and basicity. Temperature-programmed desorption (TPD) is one of the often-used methods. TPD uses desorbed species as markers of surface acidity and basicity, such as ammonia (NH_3) or carbon dioxide (CO_2), respectively. It is possible to determine the kind and intensity of acid and base sites by observing the desorption profiles at various temperatures.

Microscopy and Spectroscopy for Structural Characterization

The structural characteristics and surface makeup of carbon xerogels may be learned a lot through spectroscopic and microscopy methods. The visualisation of the carbon xerogel morphology,

including the analysis of pore structure and particle size, is made possible using scanning electron microscopy (SEM) and transmission electron microscopy (TEM). The surface functional groups and chemical bonding on the carbon xerogel surface may also be examined using spectroscopic methods including Fourier-transform infrared spectroscopy (FTIR) and X-ray photoelectron spectroscopy (XPS).

Results Comparison with Literature Data

It's crucial to contrast the acquired findings with published data in order to verify the efficacy of the oxidative treatments and gauge their influence on the surface characteristics of carbon xerogels. For assessing the effectiveness of the oxidative treatments, comprehending the changes in surface characteristics, and establishing links between the treatments and the observed alterations, published works on analogous carbon xerogel systems might be an invaluable resource.

A thorough assessment of the oxidatively treated carbon xerogels may be accomplished by combining surface area analysis, pore size distribution determination, surface acidity/basicity measurement, microscopy, spectroscopy, and comparison analysis with literature data. A greater comprehension of the structural and surface characteristics of the activated carbon xerogels, as well as their prospective applications and performance, is made possible by these characterisation approaches.

The experimental methods and processes utilised to characterise the synthesised mesoporous carbon xerogels and analyse their surface characteristics after oxidative treatment are described in the sections that follow.

Conclusion

Sol-gel condensation of resorcinol and formaldehyde, pH adjustment, gel curing, grinding, washing, drying, and carbonization are all processes in the production of mesoporous carbon xerogel. These actions result in the production of a well-defined, highly surface-aread porous structure. The resultant carbon xerogel is a flexible material with several uses.

Carbon xerogels' surfaces are activated and their surface characteristics are changed by oxidative processes such sulfuric acid, nitric acid, and air oxidation. By adding acidic functional groups, nitric acid treatment makes the surface more acidic, while sulfuric acid treatment makes the surface more basic by removing basic sites. Surface acidity is increased by air oxidation, while basicity is somewhat reduced. By adjusting their surface chemistry, these oxidative treatments allow carbon xerogels to be tailored for certain uses.

The surface properties and structural characteristics of the activated carbon xerogels are revealed by characterisation techniques such as surface area analysis, pore size distribution estimation, surface acidity/basicity measurement, microscopy, spectroscopy, and comparative analysis with literature data. These methods provide a thorough comprehension of how oxidative treatment affects the surface characteristics of carbon xerogels and serve as a foundation for future optimisation and application development.

A possible route for the creation of sophisticated materials with specific surface features is the synthesis and activation of mesoporous carbon xerogels using oxidative processes. Applications in catalysis, adsorption, and other areas where surface chemistry is important are made possible by the ability to alter surface properties.

We can realise the full potential of mesoporous carbon xerogels and contribute to the creation of creative solutions for diverse industrial and environmental problems by deepening our knowledge of their surface activation. The development of materials science and the practical use of mesoporous carbon xerogels in a variety of sectors are greatly enhanced by further study in this area.

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